A Direct Synthetic Ammonia Plant[1]

By F. A. Ernst, F. C. Reed, and W. L. Edwards

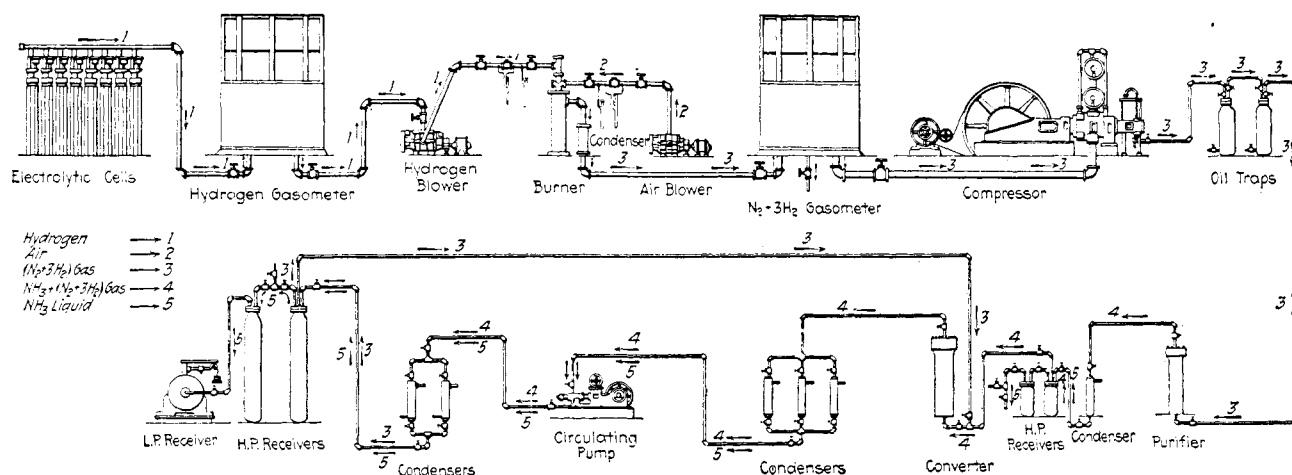Fixed Nitrogen Research Laboratory, Washington, D. C.

Figure 1—Flow Diagram of Direct Synthetic Ammonia Process

IN SPEAKING of the direct synthetic ammonia process, it has become customary to speak of the characteristic method of operation of the process as a process itself, as, for instance, "Haber Process," "Claude Process," "Casale Process," "Fauser Process," "General Chemical Process," etc. These all accomplish the synthesis of ammonia from its elements by passing a 3:1 mixture of hydrogen and nitrogen at elevated pressure and temperature over a catalytic material, and differ principally in what might be called mechanical manipulation.

The Haber process, so named because of the development of its fundamental scientific data by Fritz Haber, was the first of the direct synthetic ammonia processes to come into commercial use. The Badische Anilin und Soda Fabrik started a plant employing this process into successful commercial operation in 1913, and has since increased the total capacity as represented by two plants, both in Germany, to 300,000 metric tons of nitrogen per year. The other processes, all of later development, are represented at this time by relatively small capacity, which is for the most part widely distributed—that is, not confined to one country.

In the United States there are installations employing the "General Chemical Process," the "Casale Process," the "Claude Process" (now under construction), and a scheme of operation which has been called the "American Process."[2]

This paper will discuss in brief the design of equipment of three tons of ammonia per day capacity, for the latter type. These designs are based on the information gained from various tests and experiments conducted at the Fixed Nitrogen Research Laboratory, including the operation of its one-quarter ton ammonia unit.

Flow Diagram

The flow for this operation is shown in Figure 1. Hydrogen, here shown as from electrolytic cells, passes to the burner through a positive pressure blower with a gasometer floating on the intake line. Air is introduced into the burner through

[1] Received June 4, 1925.
[2] *Chem. Met. Eng.*, **30**, 948 (1924).

a positive pressure blower in such amount that the resulting gas after the oxygen has been burned out is a 3:1 mixture of hydrogen and nitrogen, which passes to the compressor with a gasometer floating on the line. From the compressor the gas mixture, now at 300 atmospheres, flows through the oil traps to the purifier containing an ammonia catalyst. This catalyst is operated at a higher temperature than the converter catalyst and but a relatively small amount of ammonia is synthesized, which is condensed and removed in the following condenser and receiver, taking with it those impurities of the gas which were not removed by the catalyst itself.

The resulting gas, the make-up gas to the circulatory system, is now in a very pure condition, and it is necessary to prevent contamination. This make-up gas, combining with the recirculated gas, passes into the converter, where the ammonia synthesis takes place. A portion of the ammonia in the effluent gases is then condensed out in the following condenser, and the whole mixture, nitrogen, hydrogen, ammonia gas, and liquid ammonia, passes into the circulating pump, where the liquid ammonia acts as the lubricant. It is equally as important to prevent contamination of the gases, as it is to purify the gases in the first place, and so no new material, such as oil, is introduced as a lubricant. From the circulating pump the mixture passes through the final condenser to the receiver, where the condensed ammonia is trapped out, the remaining gas passing on to meet the make-up gas for passage through the converter.

Gas Production

Gas Requirements

At 20° C. and 1 atmosphere the density of a $(N_2 + 3H_2)$ mixture is 0.02223 pound per cubic foot. There will then be required for 3 tons of ammonia $\dfrac{6000}{0.02223} = 269,900$ cubic feet of the gas mixture. If, now, it is considered that 10 per cent of the gas mixture is lost through leaks and purging, the original volume will have to be $\dfrac{269,900}{0.90} = 300,000$ cubic

feet, of which 25 per cent, or 75,000 cubic feet, is nitrogen and 75 per cent, or 225,000 cubic feet, is hydrogen.

With the composition of the atmosphere considered as

	Per cent
Nitrogen	78.14
Oxygen	20.92
Argon	0.90
Others	0.04
	100.00

there will be required $\frac{75,000}{0.7814}$ = 96,000 cubic feet of air to produce the required nitrogen. From this amount of air there will be 96,000 × 0.2092 = 20,000 cubic feet of oxygen to be disposed of, requiring 40,000 cubic feet of hydrogen. The total hydrogen requirements, therefore, will be 225,000 + 40,000 = 265,000 cubic feet measured at 20° C. and 1 atmosphere.

Electrolytic Hydrogen

Although hydrogen from any source may be used, that from the electrolysis of water will be considered here, and so only means for final purification are included.

Electrolytic cells of 5000 amperes normal operating capacity can now be secured in this country, delivering a 99.5 per cent, or better, purity of gas when operating at 60° C. at a power consumption of 140 kilowatt hours per 1000 cubic feet of hydrogen. At 2.24 volts per cell and 5000 amperes the power consumption per cell per 24 hours is 268.8 kilowatt hours. With the consumption of 140 kilowatt hours per 1000 cubic feet of hydrogen, each cell will deliver $\frac{268.8}{140}$ = 1.92 M cubic feet per 24 hours. For 265,000 cubic feet of hydrogen per 24 hours, therefore, $\frac{265}{1.92}$ = 138 cells will be required. Such cells would be of a size physically to contain each about 150 gallons of water, in which had been dissolved 435 pounds of 88 to 92 per cent potassium hydroxide. Although most cells call for distilled water, they may also be obtained recommended for operation on tap water. As operation at as high as 60 per cent over capacity causes but a small decrease in efficiency, hydrogen demands somewhat in excess of 265,000 cubic feet may be met with this bank of 138 cells. The cells operate on direct current, necessitating the conversion of alternating current, usually by means of a motor generator set, although rotary converters are sometimes used.

Nitrogen from the Air

The atmosphere presents an unlimited supply of nitrogen, which may be extracted by liquefaction methods or by burning out the oxygen with hydrogen. An apparatus for the latter method is shown in Figure 2. Hydrogen and air are admitted separately as shown and meet at a point about midway of the length of the body of the burner. Here, by means of a spark jumping from an extended lead of a spark plug, the hydrogen-oxygen mixture is exploded. The spark, of course, is necessary only on starting up, after which burning continues unaided and the nitrogen of the air together with the excess hydrogen is collected. Each volume of oxygen combines with two volumes of hydrogen and the resultant water is removed in a condenser following the burner. The gas mixture from the point of combustion flows down in to the mixing chamber and up through the cracked porcelain and shredded copper or copper turnings heated to an elevated temperature by the heat of combustion, and out. The burner operates under 2 pounds pressure, which pressure is supplied by positive pressure blowers. The hydrogen blower should be preferably liquid sealed.
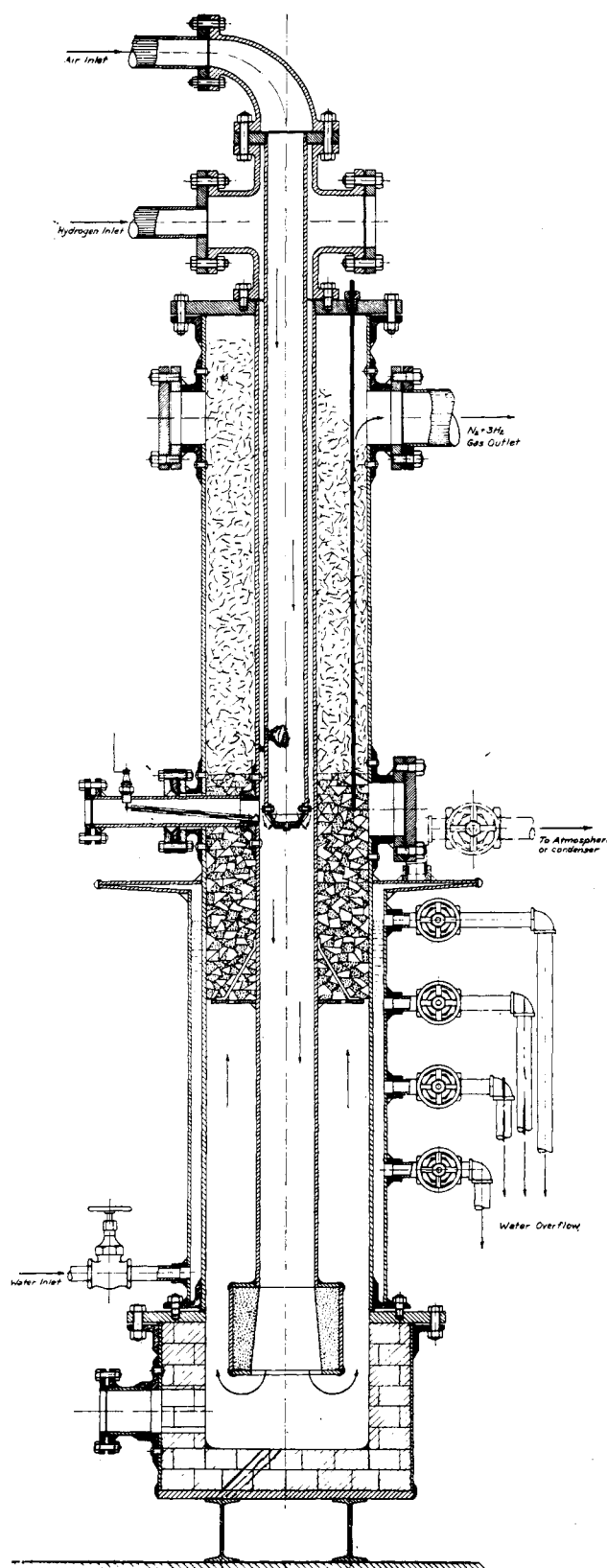

Figure 2—Hydrogen Burner

Hydrogen and air are admitted to the burner in such volumes that the effluent gas mixture is in the ratio of 3 parts of hydrogen to 1 part of nitrogen. These volumes will be in the ratio of 1 volume of air to 2.75 volumes of hydrogen.

The mixture in the burner is controlled by regulating the volume delivered by the hydrogen and air blowers. Since a definite mixture of hydrogen and air at standard conditions of atmospheric barometer and temperature is required, Venturi meters are placed before the blowers. The water manometers across the throat and entrance to the throat are provided with a gage (Figure 3) by means of which the vol-
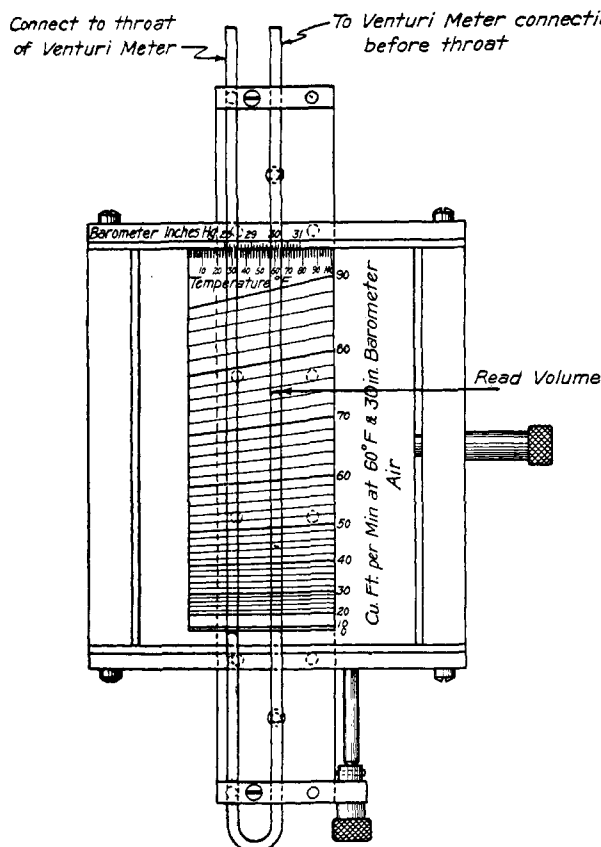

Figure 3—Standard Volume Gage

ume corrected to standard conditions is read directly. In this gage, by placing the zero of the scale at the meniscus of the left-hand water column (by adjusting with the lower screw) and by setting the existing temperature on the temperature scale on the slide into coincidence with the existing barometer on the scale on the frame (by adjusting with the thumb screw on the right), the volume as read from the scale at the meniscus of the right-hand water column is the equivalent volume of gas at standard conditions (30 inches barometer and 60 ° F.). The slide in the gage shown is graduated for air.

The volume delivered may be governed by means of by-passes or speed regulation, which may be either automatic or hand-controlled.

Compression of Gas

Gas Densities

With the catalyst developed at the Fixed Nitrogen Research Laboratory operating in this system at 300 atmospheres and 475° C., of 100 volumes of gas leaving the converter, 20 will be considered as ammonia, of which 15 will be removed, leaving 80 volumes of ($N_2 + 3H_2$) mixture plus 5 volumes of ammonia to be recirculated. Since it requires 2 volumes of ($N_2 + 3H_2$) gas to make 1 volume of NH_3, the 15 volumes of ammonia removed must be replaced with 30 volumes of make-up gas. The gas entering the converter will then be 115 volumes,

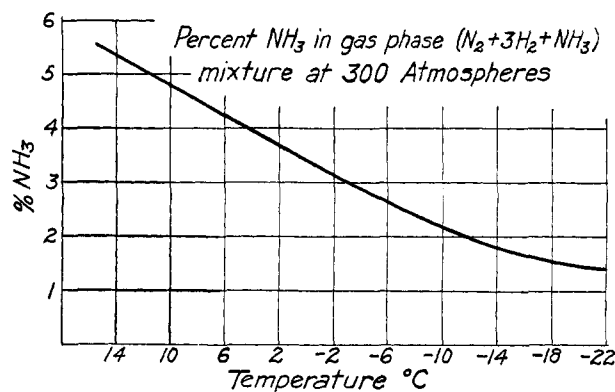made up of (80 + 30) 110 volumes of ($N_2 + 3H_2$) gas and 5 volumes of NH_3 gas.

Figure 4

The quantity of ammonia remaining as a gas after the condensers, of course, depends upon the temperature. Figure 4 shows the per cent of ammonia to be expected in the gas phase at 300 atmospheres for various temperatures.[3]

Density $= \dfrac{PV}{RT}$ (68° F. and 14.7 pounds per square inch) or

(20° C. and 1 atm.)

where P = pressure, in pounds per square foot = (14.7 × 144) = 2116.8

V = volume, in cubic feet

R = 766 for hydrogen

T = temperature, ° Abs., = 460 + 68 = 528

Then

Hydrogen $= \dfrac{2116.8 \times 1}{766 \times 528} = 0.00523$ pound per cubic foot

($N_2 + 3H_2$) = 4.25 × 0.00523 = 0.02223 pound per cubic foot

(NH_3) = 8.5 × 0.00523 = 0.04446 pound per cubic foot

Gas entering converter ($N_2 + 3H_2$) = 110 vols. = 95.6 per cent

(NH_3) = 5 vols. = 4.4 per cent
                                                    ‾‾‾‾‾‾‾‾‾‾‾‾
                                                    100.0 per cent

$= \dfrac{95.6 \times 4.25 + 4.4 \times 8.5}{100} \times 0.00523 = 0.02320$ pound per cubic foot

Gas leaving converter ($N_2 + 3H_2$) = 80 vols.

(NH_3) = 20 vols.

$= \dfrac{80 \times 4.25 + 20 \times 8.5}{100} = 0.00523 = 0.02667$ pound per cubic foot
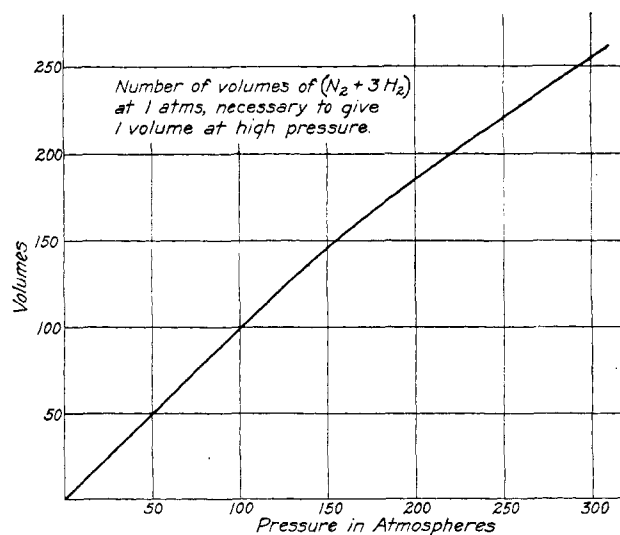

Figure 5

At these higher pressures the gases deviate quite markedly from Boyle's law, and so, instead of the volumes and densities

³ Larson and Black, *J. Am. Chem. Soc.*, **47**, 1015 (1925).

varying at 300 atmospheres, in the ratio of 1:300, they vary as 1:254. Figure 5 has been drawn from figures based on Leduc's law calculated from Amagat's *PV* curves.

Compressor Calculations

The equipment so far has been all low pressure and all more or less standard. The burner can be built up from standard piping and fittings. The compressor, the transition step from low to high pressures, is, for 300 atmospheres, a piece of equipment which may be secured from several compressor manufacturers in this country. The remainder of the high-pressure equipment is for the most part, at the present time, special. Several equipment manufacturers, in addition to a number of operating concerns interested in the production of synthetic ammonia, have secured sets of prints of the designs of a 3-ton plant, of which the accompanying illustrations are a part, and it is expected that soon most of this equipment will also be procurable as more or less standard machinery.

The following outline of calculations for a 4-stage compressor will give an idea of the effect of the variations from Boyle's law on the proportioning of stage cylinder diameters for a $(N_2 + 3H_2)$ mixture discharging at 300 atmospheres and considering 4 stages of compression:

Pressure ratio, $R = \sqrt[4]{\dfrac{300}{1}} = 4.16$

P_a = pressure of atmosphere = 14.7 pounds per square inch

P_1, P_2, P_3, P_4 = pressure at end of 1st stage, 2nd stage, etc.

T_a = temperature of atmosphere = 60° F.

T_1, T_2, T_3, T_4 = temperature at end of 1st stage, 2nd stage, etc.

$P_1 = R \times P_a = 4.16 \times 14.7 = 61.2$ pounds per square inch = 4.16 atm.

$P_2 = R \times P_1 = 4.16 \times 61.2 = 255$ pounds per square inch = 17.3 atm.

$P_3 = R \times P_2 = 4.16 \times 255 = 1060$ pounds per square inch = 72.0 atm.

$P_4 = R \times P_3 = 4.16 \times 1058.3 = 4410$ pounds per square inch = 300 atm.
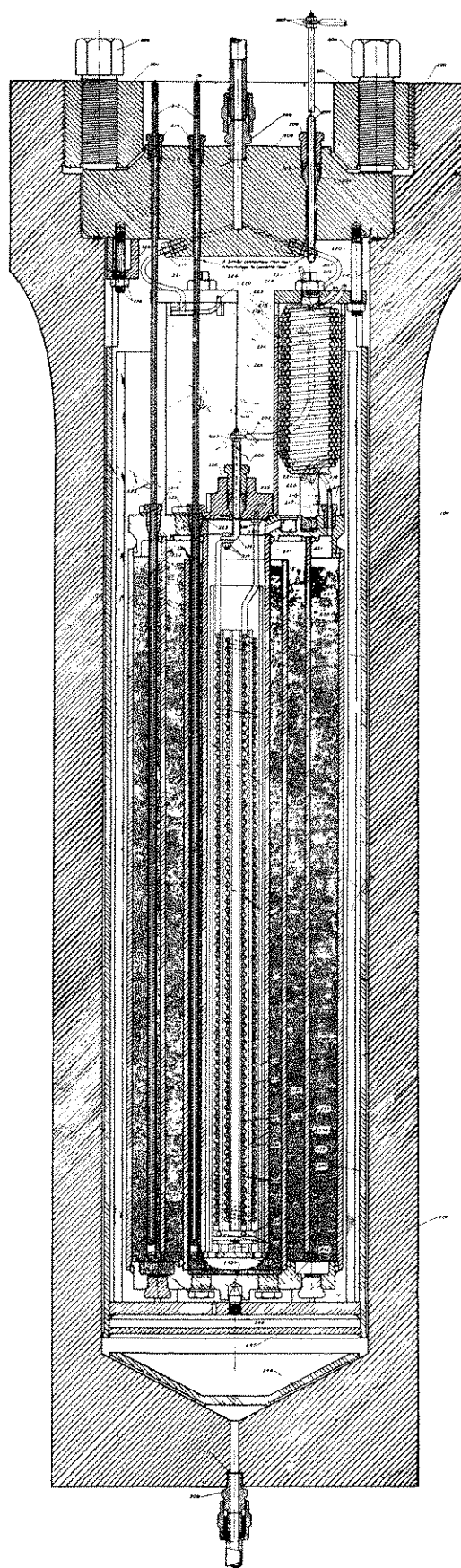
From Figure 5 it is found that the gas volumes are for
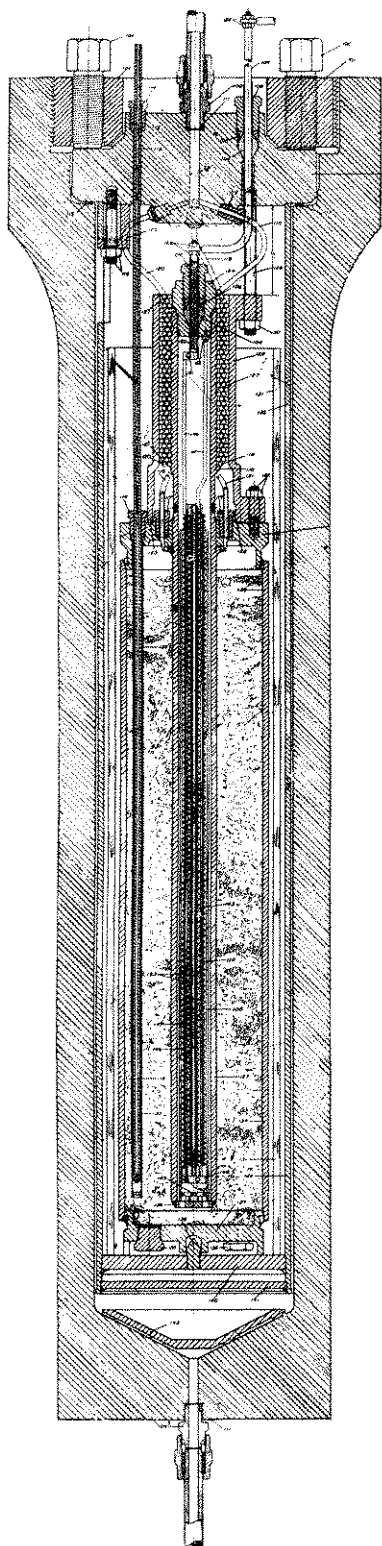

Figure 6—Purifier

Figure 7—Converter

$$
\begin{aligned}
4.16 \text{ atm.} &= \quad 4.16 \\
17.3 \text{ atm.} &= \quad 17.3 \\
72.0 \text{ atm.} &= \quad 70.7 \\
300.0 \text{ atm.} &= 254.5
\end{aligned}
$$

T_1, T_2, T_3, T_4 before intercooler $= \quad 4.16^{0.29} \times 520 = 786°$ Abs.
$= 326°$ F.
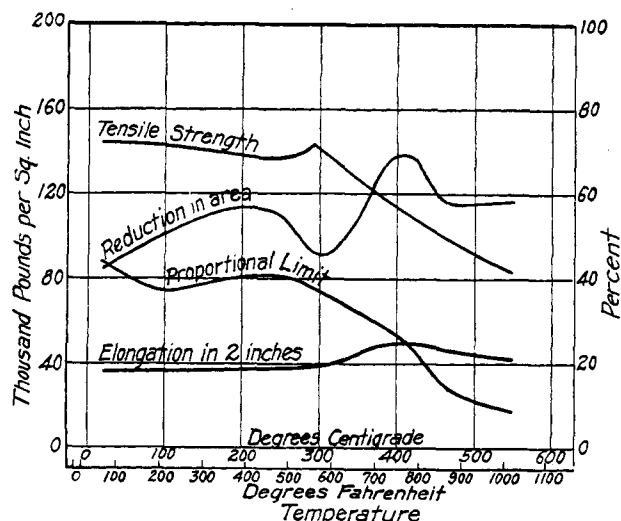
Tensile properties at elevated temperatures of a chromium vanadium steel.

Figure 8

It will be assumed that the inter-coolers and after-cooler bring the temperature of the gas in each stage down to the temperature of the inlet. Then, if Boyle's law held, the diameter of the cylinders for the different stages would vary as $\sqrt{R}$, and the work of compression would be the same for each stage. Since the gas does not obey Boyle's law, and since it is desired to equalize the work of the several stages, it will be necessary to proportion the cylinder according to a new ratio, R', based on the volumes for the pressures shown above. The effect of this will be to increase the diameter of the last two stages. If such precautions for equalizing the work were not made, the pressure ratio of the first stages would have been increased, thereby lowering the volumetric efficiency and increasing the temperature rise in that stage. The corrected ratios will then be

$$
\begin{aligned}
R'_1 &= R'_2 = R = 4.16 \\
R'_3 &= \frac{70.7}{17.3} = 4.08 \\
R'_4 &= \frac{254.5}{70.7} = 3.60
\end{aligned}
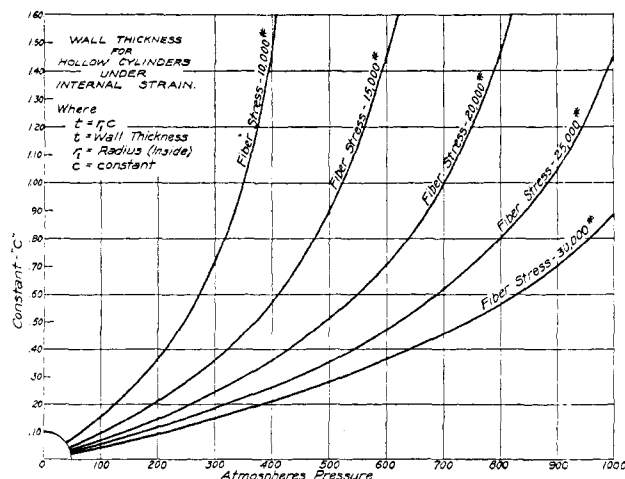$$

Figure 9

For a delivery of 215 cubic feet of gas per minute at standard conditions, 30 inches barometric pressure and 60° F., a piston speed of 380 feet per minute, single acting, will be considered. A clearance volume of 8 per cent would give a volumetric efficiency of $[1 - 0.08 \ (4.16 - 1)] = 75$ per cent. To provide for leakage at packings, etc., for these higher pressures, a volumetric efficiency of 70 per cent will be assumed. The compressor should be equipped with clearance pockets, which will take care of over-capacity resulting from a possible better volumetric efficiency. The piston diameters, then, are:

$$
\frac{\text{Piston area (less rod)} \times 380}{144 \times 2} = \frac{215}{0.70}
$$

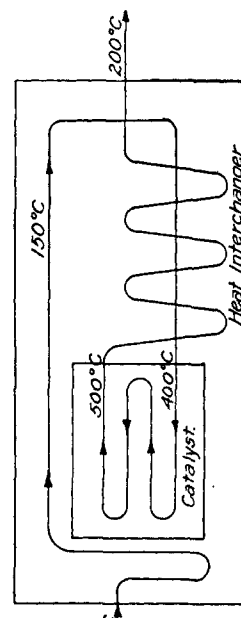Piston area (less rod) $= \dfrac{215 \times 144 \times 2}{380 \times 0.70}$

$= 233$ square inches

Fi;ure 10

Area of 2.375 inch piston rod $= 4.43$ square inches
Total area of piston 237.43 square inches

Diameter of 1st cylinder $= d_1 = \sqrt{\dfrac{237.43}{0.25 \times 3.1416}} = 17.4$ inches, say 17.5 inches

Diameter of 2nd cylinder $= d_2 = \sqrt{\dfrac{233}{\dfrac{0.25 \times 3.1416}{4.16}}} = \dfrac{17.2}{\sqrt{4.16}}$

$= 8.43$ inches, say 8.5 inches

Diameter of 3rd cylinder $= d_3 = \dfrac{8.43}{\sqrt{4.08}} = 4.17$ inches, say 4.25 inches

Diameter of 4th cylinder $= d_4 = \dfrac{4.17}{\sqrt{3.60}} = 2.20$ inches, say 2.25 inches

With an assumed stroke of 17 inches, the revolutions per minute will be:

$$
\text{R. p. m.} \times \frac{17}{12} \times 2 = 380 \qquad \text{R. p. m.} = 135
$$

High-Pressure Equipment

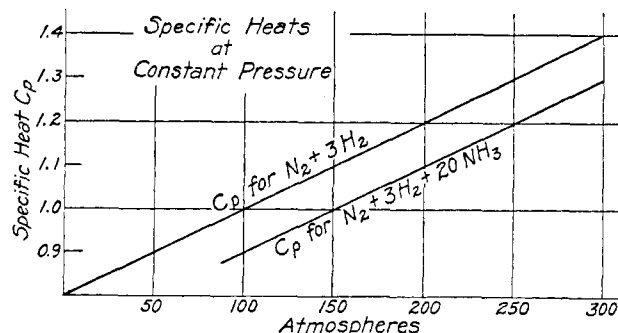The remainder of the process will be at the relatively high pressure of 300 atmospheres.

Figure 11

Purification

Figure 6 shows the purifier. As the general arrangement and construction is the same as for the converter described later, only the flow of the gas through the purifier will be outlined. The cool gas entering at the bottom travels up through the narrow annular space in immediate contact with the inner surface of the pressure-sustaining shell, to the top of the shell and through a series of holes in the baffle cylinder, as shown in the upper left-hand corner of the drawing. The flow then continues down around the outside of the coiled tubing of the heat interchanger, where it picks up the heat of the hot outgoing gases within the tubes, through the small openings at the bottom of the exchanger, into the heating element well, down around the heating element, and into the catalyst chamber. The gas, after passing up through the catalyst mass, passes out of this chamber through the inside of the tubes of the coils of the heat interchanger, where it gives up its heat to the incoming gas, and finally, relatively cool, out through the common take-off in the head. The condenser and high-pressure receivers with weighing devices are as shown and described later.

Converter

ASSEMBLY—Figure 7 shows the converter. The flow of gas is similar to that of the purifier, except that the catalyst is distributed in three annular spaces, and owing to the larger volume of gas there is increased heat exchanger capacity made up of three sets of coils similar to that of the purifier and operating in parallel.

By distributing the catalyst in this fashion the heat gradient is flattened out, decreasing in intensity the area of relatively extreme heat. The gas with the greatest capacity for

The buttress threads of the ring and shell are interrupted, so that in dismantling it is only necessary after loosening the bolts to give the ring a one-twelfth circumferential turn in order to disengage the threads and relieve the head so that it may be lifted out.

The catalyst space required for the conditions as previously set forth, and assuming a 20,000 space velocity is

$$\frac{\frac{115}{30} \times \frac{270,000}{24}}{20,000} = 2.16, \text{ say } 2.25 \text{ cubic feet}$$

Space velocity as here used is the ratio of volume of gas passing into the converter per hour per unit volume of catalyst.

The catalyst chamber is formed of steel cylindrical walls welded to the steel heads. In both the upper and lower heads are plugs through which catalyst may be loaded and removed. A thermocouple in the wells in the innermost and outermost catalyst spaces will permit temperatures of the gas to be taken as it strikes and leaves the catalyst, as well as to indicate the temperature of the catalyst along the length of these annular rings. The heating element, one lead of which is grounded to the converter itself, is contained in the interior of the catalyst chamber. It is of nichrome wire wound over porcelain insulators. The take-in lead of copper enters the head through a soapstone insulating bushing which when compressed by the gland is also a gas seal. A
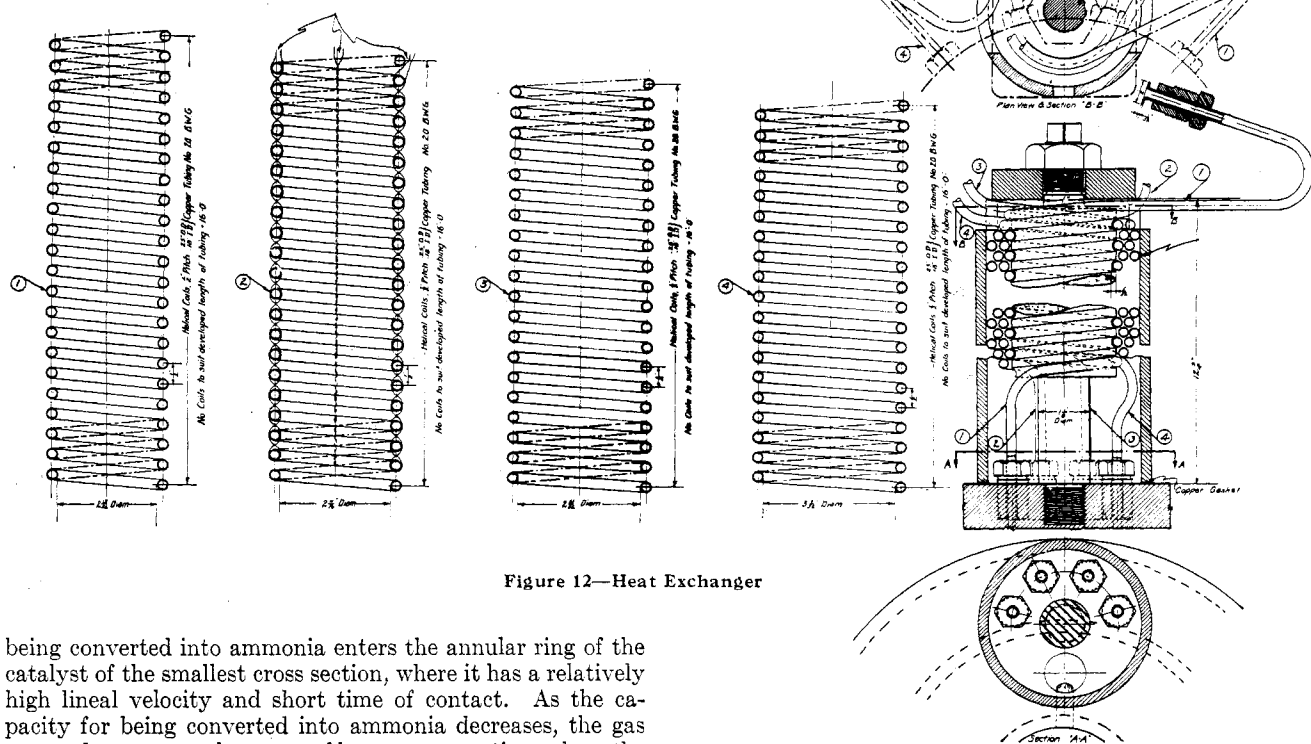

Figure 12—Heat Exchanger

being converted into ammonia enters the annular ring of the catalyst of the smallest cross section, where it has a relatively high lineal velocity and short time of contact. As the capacity for being converted into ammonia decreases, the gas enters the next annular space of larger cross section, where the lineal velocity is decreased and time of contact increased, and so from this to the third and still larger annular space.

The pressure-sustaining vessel, the material and stresses of which are discussed later, is made up of the steel shell closed by means of a head made gas-tight with a flat copper gasket, and held in place by the ring and bolts as shown.

Pyrex glass heat reflector here, as in the case of the purifier, surrounds the catalyst chamber. The heat interchangers, of which there are three similar to the one of the purifier, operate in parallel. They consist of four copper coils screwed together over a spindle and jacketed by a steel cylinder.

Each coil has a separate connection to the head and to the catalyst chamber made by upsetting and rolling out a flare on the ends of the tubes. This flare acts as the gasket between the tightening gland and the head. The catalyst chamber, heater, pyrometer wells, and heat exchangers are assembled as a unit and catalyst loaded. With the head hanging from a chain fall or tackle by a ring bolt screwed into the gas outlet tap, this unit is secured to the head by means of three bolts shown in the upper right (Figure 7), through the top cap of the heat exchangers made heavy for this purpose. This assembly is then lowered into the steel baffle
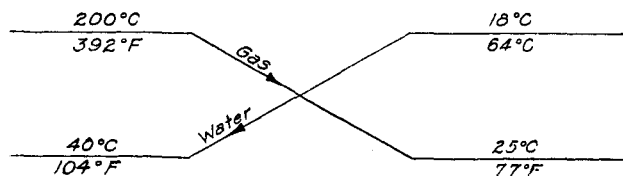

Figure 13—Temperature Assumptions, Water-Cooled Condenser

shell containing the Pyrex glass cylinder and is centered by means of the pin at the bottom. The cylinder is then secured to the head by means of the clamps and bolts shown in the upper left. The bolts, upper right, which secured the inner works to the head, are then removed, so that the weight is finally carried by these clamps and bolts. The weight of the catalyst chamber then rests on the plate forming the false bottom to the baffle cylinder taking the strain off of its welded joints. This entire assembly is now ready to be placed into the converter shell.

In the converter, as in the case of the purifier, the entering cool gas passes in immediate contact with the inner periphery of the pressure-sustaining vessel, thus tending to keep down the temperature of the steel. This scheme, used in most all the later synthetic ammonia processes, is also used in many other operations and was suggested several years ago by the Geophysical Laboratory when work was started on high pressure synthesis of ammonia at the Fixed Nitrogen Research Laboratory.

FABRICATION OF SHELL—The importance of keeping the temperature of the steel below 250° C. can be appreciated from the curves of Figure 8,[4] where it is seen that the proportional limit drops off rapidly from that point on.

The ideal composition of steel for conditions such as are met in an ammonia converter is a chrome vanadium steel in which the ratio of carbon to chromium is 1:8. It has been found, however, that no significant improvement is obtained in tensile properties by having over 2.5 per cent chromium. A steel that would be considered good is one containing 2.5 per cent chromium, 0.18 to 0.20 vanadium, and 0.30 to 0.35 carbon, for such parts (shell and head) as come in contact with the hot reducing ammonia gases. The ring and bolts may be of a 3 per cent nickel steel. The bolts should be treated for an elastic limit of 90,000 pounds per square inch, and the shell, head, and ring should show an elastic limit of 65,000 pounds per square inch.

In the forging such as that required for the converter shell, cracks, porosity, or other imperfections sometimes appear along the longitudinal axis. To take care of this possibility holes for 2-inch bolts for a cover plate 6.75 inches in diameter across the center line of the gasket were tapped into the base of the shell. This particular shell, however, proved on test to be perfect and the plate was unnecessary.
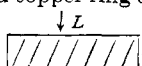
STRESS CALCULATIONS—The internal dimensions of the converter are, of course, fixed by the outside dimensions of the inner parts. It is necessary, then, to compute the external dimensions.

[4] French, *Bur. Standards, Tech. Paper* **205.**

For figuring thick-walled tubes, the use of a formula $r_2 = r_1 \left(\dfrac{3T + R}{3T - 4R} \right)^{1/2}$, worked up in 1880 by Clavarino as a modification of the formula deduced by Lamé in 1833, is good practice. Here r_2 = outside radius, r_1 = inside radius, T = tangential stress, R = unit internal pressure.

Figure 9 is a set of curves worked up from Clavarino's formula for pressures up to 1000 atmospheres. From these for a cylinder of 16 inches internal diameter, to be subjected to an internal pressure of 300 atmospheres, it is found that for an allowable stress of 15,000 pounds per square inch, the wall thickness $t = r_1 C = 8 \times 0.35 = 2.80$ inches. A wall thickness of 3 inches was used. To check this by a simplified Lamé's formula $\left(\dfrac{r_2}{r_1} \right)^2 = \dfrac{T + R}{T - R}$, the stress T is found to be $\left(\dfrac{11}{8} \right)^2 = \dfrac{T + 4400}{T - 4400}$ or $T = 14,300$ pounds per square inch.

The gasket is a flat, unconfined copper ring 0.125 inch thick.

The theoretical normal load, L ↓L   //////// ←—P, required to keep the gasket from blowing and to seal the internal gas pressure from leaking out, with friction F as 0.15, would be the gas pressure against the edge of the gasket divided by
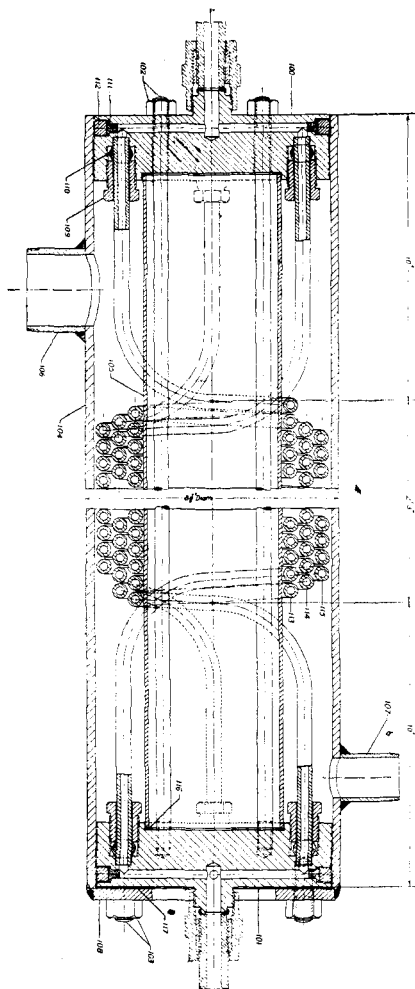

Figure 14—Condenser (Water-Cooled)

double friction, $2F$, or $L = \dfrac{P}{0.3}$. It has been found experimentally that a constant $C = 2.5$ must be introduced for regular "close finish" machining, because of the practical impossibility of machining absolutely perfect the surfaces
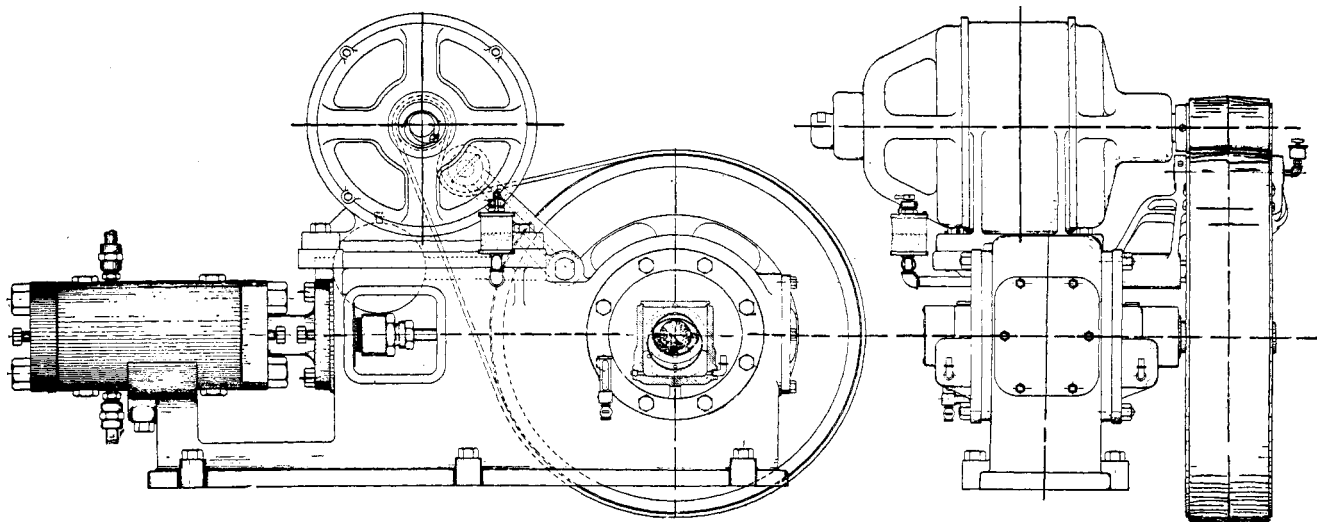
Figure 15—Circulating Pump

between which the gasket is squeezed. This formula then becomes $L = \frac{P}{0.3} \times 2.5$. To form a positive gas seal, a small V groove, equivalent to a continuous tool mark, is cut into the surfaces between which the gasket is compressed. The width of the gasket is such that the load applied by means of the bolts is at least 60,000 pounds per square inch of gasket face. Commercial copper of this thickness flows at 60,000 pounds per square inch. The groove is then filled with the flowing copper and a cross section of the gasket appears

thus . The gasket for the converter

is figured as follows:

$$L = \frac{4400 \times 0.125 \times 2.5}{2 \times 0.15} = 4580 \text{ pounds per inch of length i. d.}$$

To this must be added the load required to counterbalance the internal gas pressure against the head as the gasket is made up before pressure is put on the vessel.

$$\frac{16.375^2 \times \frac{\pi}{4} \times 4400}{16.375 \times \pi} = 18,000 \text{ pounds per inch of internal length of gasket}$$

The total load per inch length of gasket then becomes $4580 + 18,000 = 22,580$ pounds and the width of gasket is $\frac{22,580}{60,000} = 0.38$ inch. The width of the gasket will adjust itself to any applied load greater than 60,000 pounds per square inch by flowing, thus increasing its surface area and
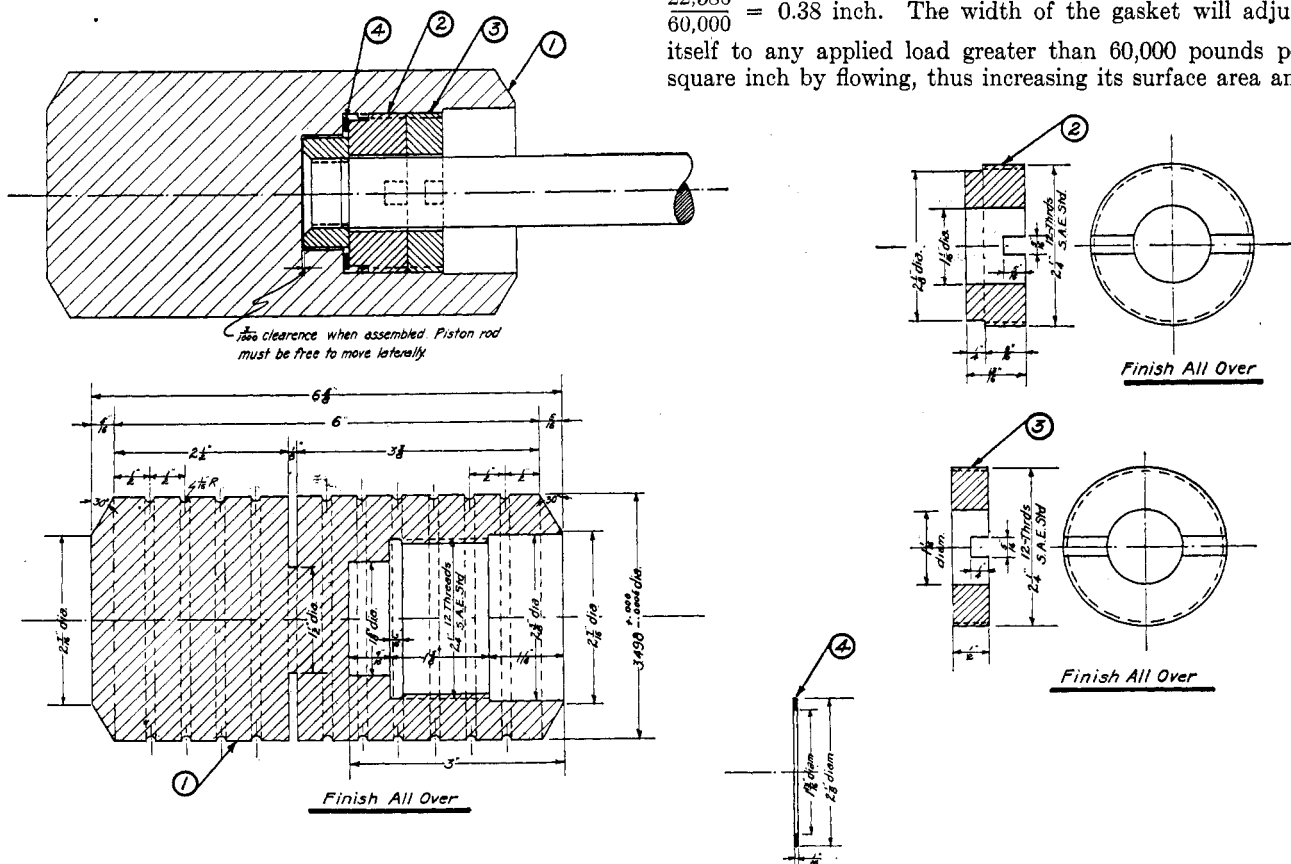

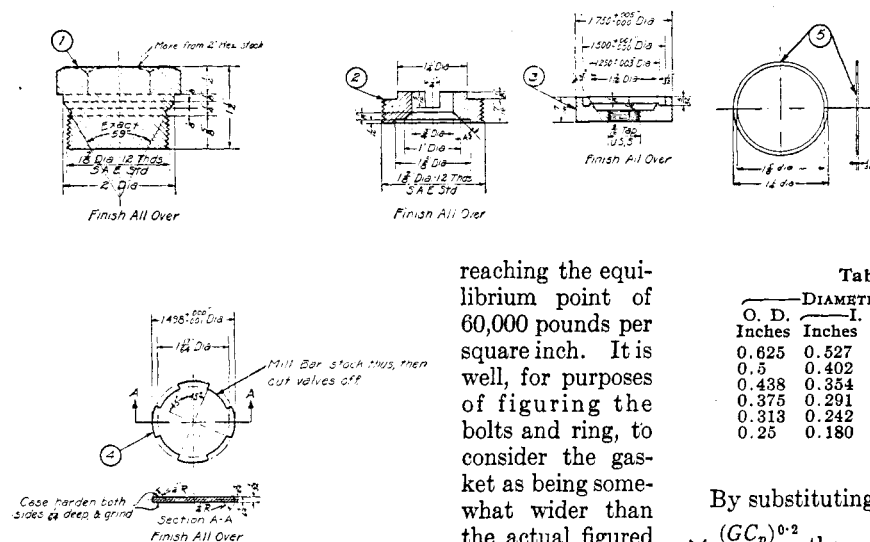**Figure 16—Circulating Pump Piston**

Figure 17—Circulating Pump Valves

$$L = 1290 \, D \left(\frac{L}{DN}\right)^{0.2} \text{ feet}$$

For values of $N = 1$, 2, 4, 6, 8, and 12, and various values of D (all commercial sizes for copper tubing), L is as shown in Table I.

Table I—Length of Tubes, in Feet

——DIAMETER——								
O. D. Inches	I. D. Inches	Meters	$N=1$	$N=2$	$N=4$	$N=6$	$N=8$	$N=12$
0.625	0.527	0.01339	41.0	35.7	32.6	28.5	26.9	24.9
0.5	0.402	0.01021	33.0	31.1	28.6	26.4	24.8	23.0
0.438	0.354	0.00898	29.7	25.9	22.5	20.8	19.6	18.1
0.375	0.291	0.00739	25.4	22.1	19.3	17.8	16.8	15.4
0.313	0.242	0.00616	22.0	19.1	16.7	15.4	14.5	13.4
0.25	0.180	0.00457	17.2	15.0	13.0	12.0	11.3	10.5

By substituting for $\frac{L}{D}$ in Equation 2, its value $129 \times \frac{T}{t_m}$ $\times \frac{(GC_p)^{0.2}}{DN}$ the pressure drop expression then becomes $\Delta P =$ $(0.0001 + 0.0000258 \, \frac{T}{t_m} \times C_p^{0.2})\frac{G^2}{N^2 D^4 \rho}$ kg. per sq. cm., in which $T = 300$, $t_m = 71$, $C_p = 1.3$, $G = 0.149$, $\rho = 50$.

reaching the equilibrium point of 60,000 pounds per square inch. It is well, for purposes of figuring the bolts and ring, to consider the gasket as being somewhat wider than the actual figured width. If, now, the width is taken as 0.5 inch, the gasket area from the new diameter of 16.375 + 0.5 = 16.875 inches is 16.875 × π × 0.5 = 26.5 square inches. At 60,000 pounds per square inch of surface area, the bolts must be able to stand 26.5 × 60,000 = 1,590,000 pounds collectively. If fourteen bolts 2.25 inches in diameter with buttress threads are considered, each bolt must take $\frac{1,590,000}{14}$ = 114,000 pounds, equivalent to $\frac{114,000}{3.24}$ = 35,200 pounds per square inch compression at root of thread. This stress is transmitted by means of the bolts to the ring and from this into the shell.

HEAT INTERCHANGE—The method of calculating the heat interchange system is that developed by the Cryogenic Laboratory of the U. S. Bureau of Mines from Nusselt's original work. It is known as the pressure drop method. The method of procedure is to assume the temperature drop and calculate from this the lengths of tubes of various diameters required. With these lengths the pressure drops are determined and from these is selected the number of tubes of a certain diameter and length to give what may be considered a reasonable pressure drop.

The formulas for this calculation are, for mean temperature difference

$$t_m = 129 \times T \times \frac{D}{L} \times \left(\frac{GC_p}{DN}\right)^{0.2} \tag{1}$$

and for pressure drop

$$\Delta P = \left[0.0001 + 0.0000002 \times \frac{L}{D} \times \left(\frac{DN}{G}\right)^{0.2}\right]\frac{G^2}{N^2 D^4 \rho} \tag{2}$$

in which
ΔP = pressure drop through tubes, in kg. per sq. cm.
t_m = mean temperature difference, in ° C.
T = temperature rise or drop, in ° C.
D = inside diameter of tubes, in meters
L = length of tubes, in meters
N = number of tubes
G = mean flow of gas, in kg. per second
C_p = mean specific heat of gas, in kg. cal. per kg.
ρ = mean density of gas, in kg. per cu. m.

Figure 10 shows the thermal assumptions for the converter. Figure 11 is a curve for specific heat at constant pressure worked up from the data contained in "Physical and Chemical Data of Nitrogen Fixation," by the Nitrogen Products Committee of the British Ministry of Munitions.

In Equation 1, $T = 300$, $t_m = 71$, $G = 0.149$, $C_p = 1.3$. By substituting these values and converting from meters to feet, Equation 1 becomes

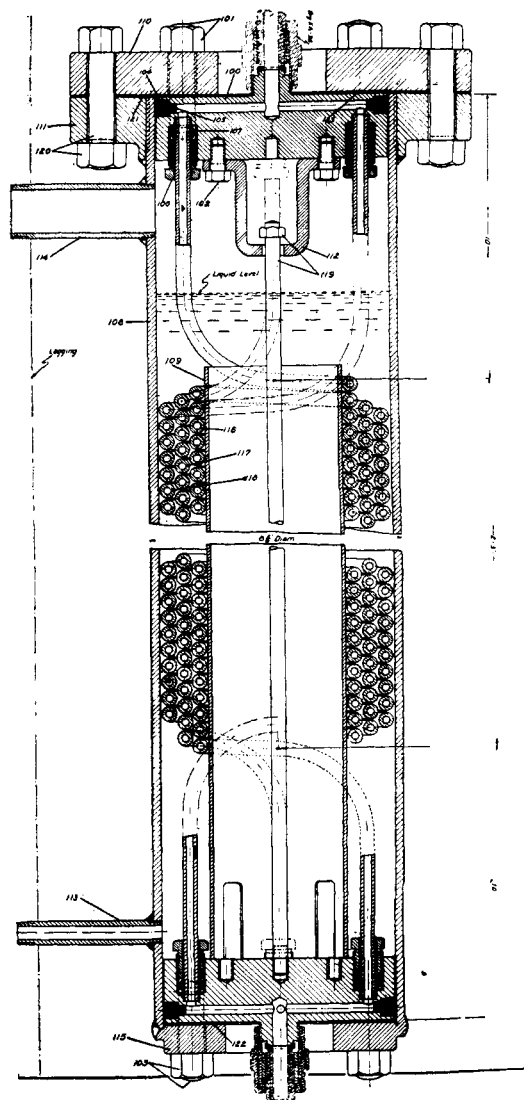Figure 18—Condenser—Direct Ammonia Expansion

ΔP in pounds per square inch for the values of N and D as of Table I are shown in Table II.

Table II—Pressure Drop, ΔP, in Pounds per Square Inch

DIAMETER								
O. D. Inches	I. D. Inches	Meters	$N=1$	$N=2$	$N=4$	$N=6$	$N=8$	$N=12$
0.625	0.527	0.01339	43	11.0	3	1.2	0.7	0.3
0.5	0.402	0.01021	126	36	8	3.5	2.0	0.4
0.438	0.354	0.00898	211	53	13	5.9	3.3	1.4
0.375	0.291	0.00739	463	116	29	12.8	7.2	3.3
0.313	0.242	0.00616	950	238	59	26	14.8	6.7
0.25	0.180	0.00457	3140	785	196	87	49	21.7

For the converter heat interchanger, twelve tubes, each 0.25 inch o. d. No. 20 stubs gage were used, giving, as shown in the tables, a calculated pressure drop of 21.7 pounds for tubes 10.5 feet in length. The interchange system was made up in three parallel nests, each as shown in Figure 12, consisting of four tubes each 16 feet long, giving a total pressure drop of approximately 25 pounds.

Condensers (Water Overflow Type)

The condensers after both the purifier and converter are of the overflow water type. The temperature assumptions are as shown in Figure 13.

$$\text{Here } t_m = \sqrt{288 \times 13} = 61° \text{ F.}$$
$$\text{Temperature drop} = 392 - 77 = 315° \text{ F.}$$
$$\text{Weight of gas} = 1000 \text{ pounds per hour}$$
$$C_p = 1.3$$
$$\text{Heat transfer} = 1.3 \times 315 \times 1000 = 410,000 \text{ B. t. u. per hour}$$

By actual test, a heat transfer of 150 B. t. u. per square foot per ° F. per hour was found conservative for 0.5 inch o. d. $\times$ 0.25 inch i. d. steel tubing. Using this figure, the number of square feet of cooling surface is found to be $\dfrac{410,000}{150 \times 61}$ = 45 square feet.

If 0.25 inch i. d. tubing is considered, there will be required $\dfrac{45}{\dfrac{0.25 \times \pi \times 1}{12}}$ = 688 lineal feet. For nine tubes in parallel,
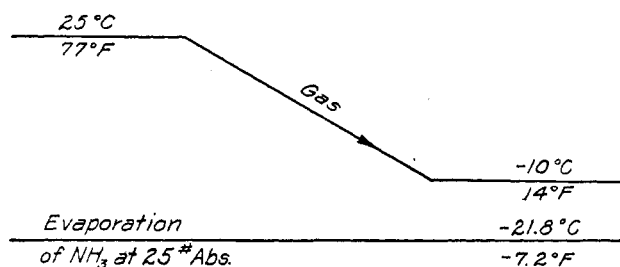

Figure 19—Direct Expansion Condenser

each tube would be $\dfrac{688}{9}$ = 77 feet long and the mean velocity of the gas through these tubes would be 16 feet per second as follows: mean temperature of gas = $\sqrt{392 \times 77}$ = 173° F. = 633° Abs.; mean volume = $\dfrac{633}{852} \times 0.0678$ = 0.0493 cubic foot per second. The area of nine 0.25 inch i. d. tubes = 0.00307 square foot. The mean velocity of the gas, therefore, will be $\dfrac{0.0493}{0.00307}$ = 16 feet per second. The quantity of cooling water required will then be 40° F. $\times$ weight of water per hour = 410,000 B. t. u., or weight of water per hour = 10,250 pounds = 22 gallons per minute total. For the condensing system after the converter this quantity of equipment was arranged in three parallel units, each as shown in Figure 14,
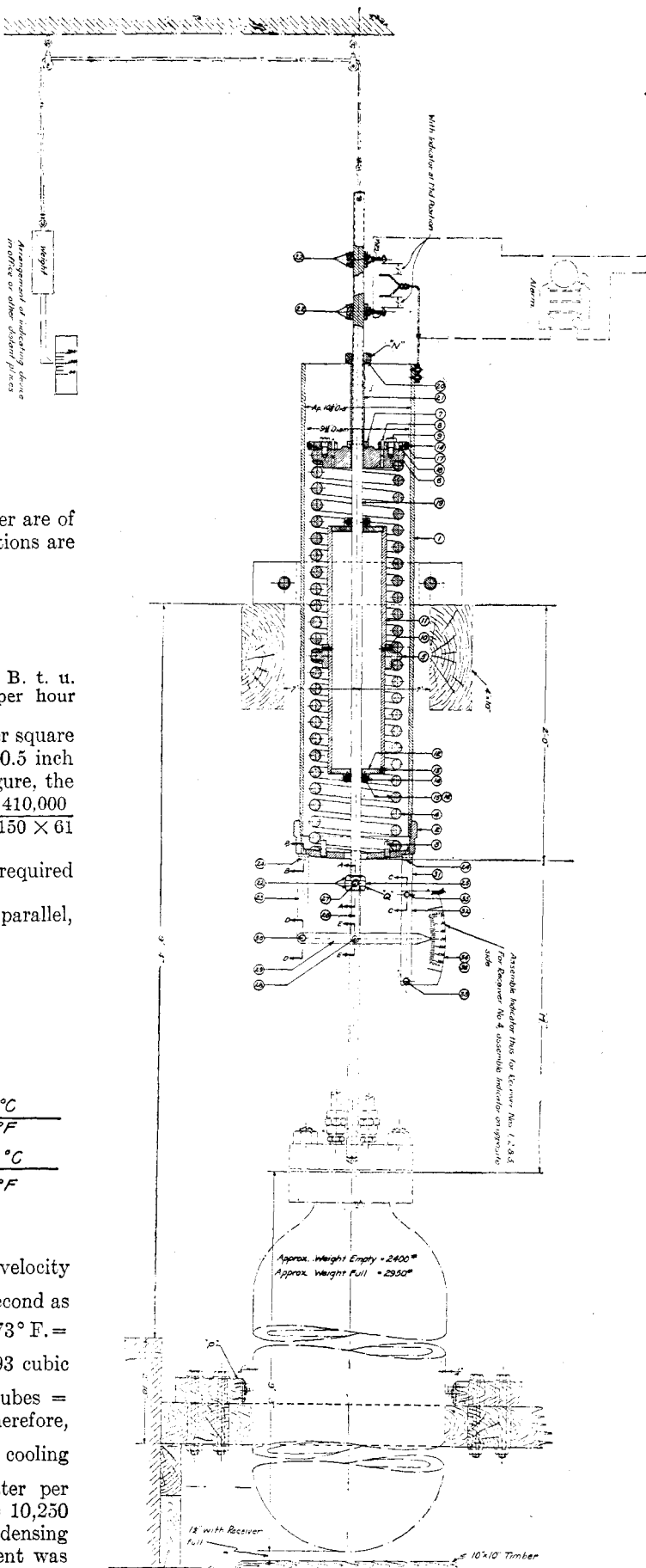

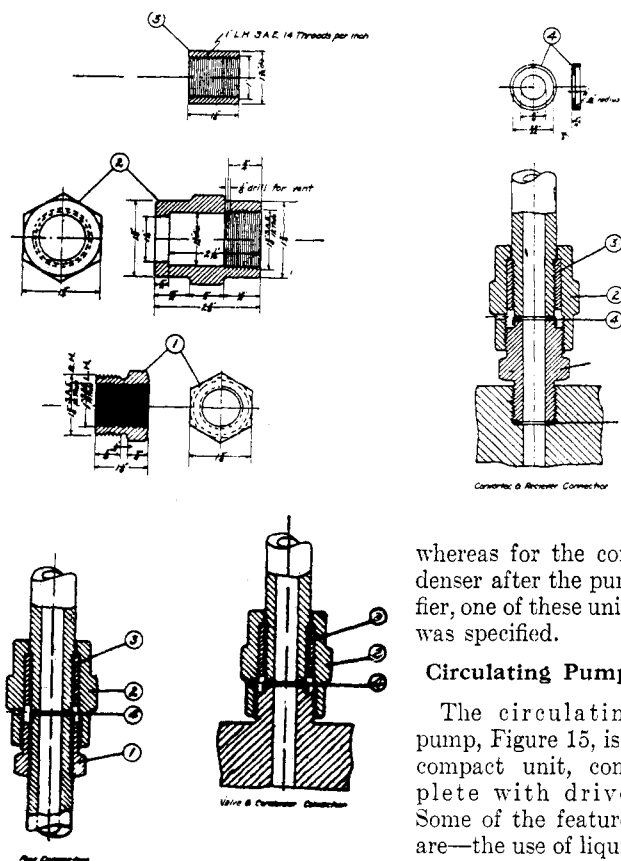
Figure 20—Liquid Ammonia Receivers with Weighing Device

inder, suction on bottom and discharge on top, that they are sealed with liquid ammonia.

The piston rod packing consists of a cast-iron bushing pressed into the cylinder head. The clearance between the bushing and the accurately ground and hardened piston rod is extremely small. The metallic packing consists of alternate rings of special Babbitt metal and steel diamond-shaped rings. By screwing down on the packing gland nut the diamond shape of the steel rings forces the Babbitt metal out against the piston rod. The cage forms a vent, from which the leakage is conducted, through a scrubber to remove the ammonia, to the mixed gas holder. This gas containing ammonia, expanding from 300 atmospheres in the cylinder of the pump to holder pressure at the cage, keeps the packing and cylinders below room temperature.

Close clearance between the piston rod and packing is permissible because of the floating type of piston rod. The flexible connection to the piston and cross head allows the piston rod to move laterally with respect to the piston and cross head, so that neither the piston nor cross head can in
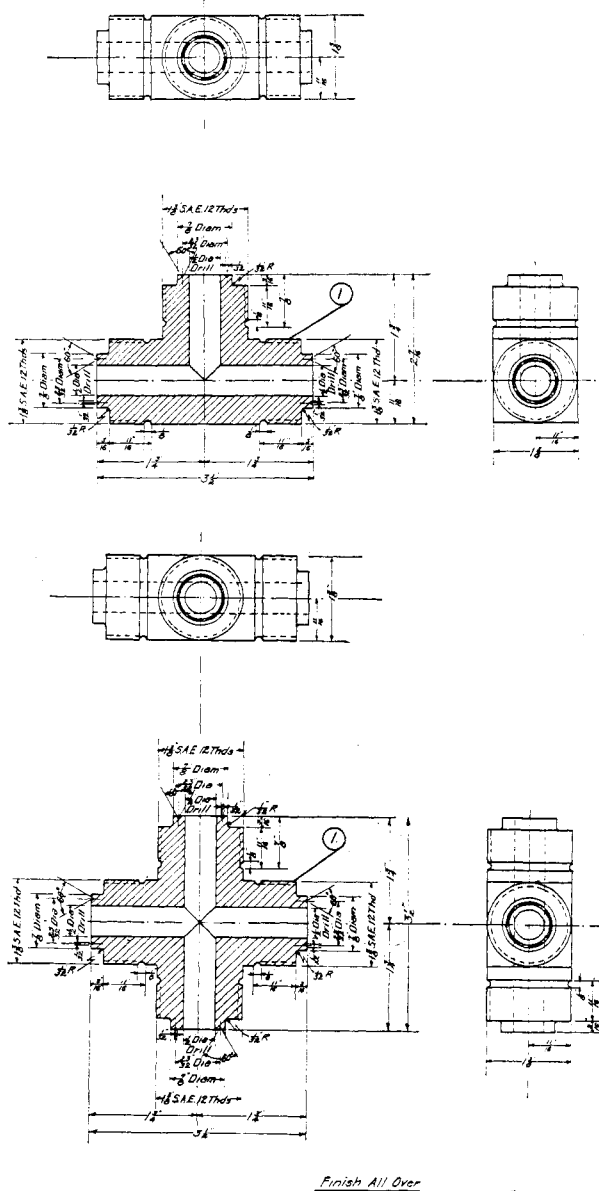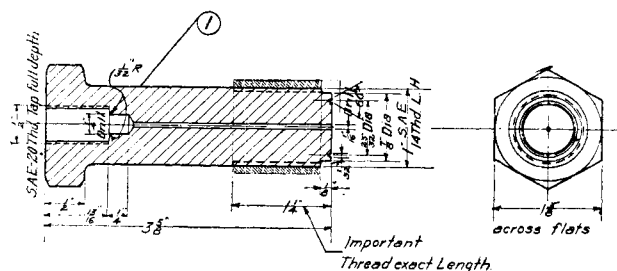

Figure 21—Union Connections

whereas for the condenser after the purifier, one of these units was specified.

Circulating Pump

The circulating pump, Figure 15, is a compact unit, complete with drive. Some of the features are—the use of liquid ammonia (a product of the plant) as a lubricant, the plate valves, the piston rod packing, and the new floating type of piston.

The only lubricant used in the pump cylinder is the ammonia liquefied in the condensers immediately before the pump. Piston rings are not used, but the piston (Figure 16), ground to a close clearance, is provided with circular grooves which when filled with liquid ammonia form a seal against leakage by the piston. The deep groove in the piston is to minimize any tendency of the piston to warp.

The valve, Figure 17, is especially adapted to high pressure and small volume compressors and pumps. It is a flat plate type of relatively large diameter, thus requiring but a small

Figure 22—Special Fittings

lift, $1/64$ inch, permitting of a very narrow seat, $1/32$ inch, approaching line rather than surface contact for sealing the gas. This narrow seat also reduces the pressure differential required for opening the valve, thus saving compression and almost eliminating the troubles of grit lodgment on the seat. The valve breathes rather than pounds in contrast to the ordinary plate valve. The valves are so arranged in the cyl-

Figure 23—Regular Fittings

any way bind the piston rod. Adjustment is made by means of laminated copper shims.

The frame is extended to support the cylinder and is bored at that point at the same time the cross head guide is bored, thus assuring perfect alignment.
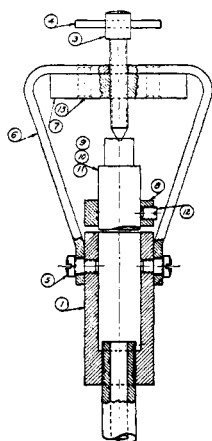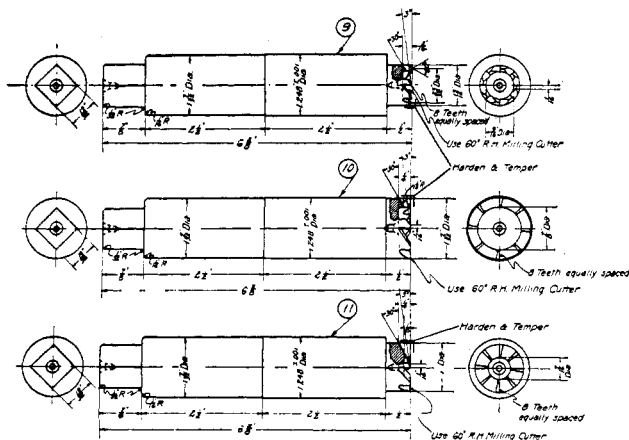

Figure 24—Pipe Joint Finishing Tool

The bearings are provided with removable bushings and are ring-oiled. The crank pin is self oiling, while the wrist pin is oiled by a sight feed lubricator. The gaskets are the nonconfined, flat type flowing into the V grooves as used on the converter. The cylinder at the head end is bored slightly larger than at the piston end, so that the cylinder can be rebored without changing heads.

Condenser (Direct Expansion)

Final condensing is accomplished in two parallel, direct ammonia expansion type condensers (Figure 18). As the expanded ammonia is to be taken direct to the absorption tanks of either the aqua system or the ammonia salt plant, expansion will take place at 25 pounds absolute. The corresponding saturated temperature of ammonia for this pressure is $-7.2°$ F. Thermal conditions as shown in Figure 19 will be assumed.

Mean temperature, t_m = $\sqrt{84 \times 21}$ = 42° F.
Weight of gas = 1000 pounds per hour
Heat drop = $1.3 \times 1000 (77-14)$ = 81,900 B. t. u.

The heat transfer in this case will be half that for the previous case, but the temperature differential is only about half as great, so that the rate of heat transmission expected would be somewhat less.

$$\frac{81,900}{75 \times 42} = 26 \text{ square feet}$$

Two condensers equivalent in exchange surface to the water-cooled condensers having a surface of $15.7 \times 2 = 31.4$ square feet will be used.

Latent heat of ammonia at 25 pounds absolute and $-7.2°$ F. = 577.8 B. t. u. per pound. The heat lost in cooling ammonia from 14° F. to $-7.2°$ F. considering the specific heat of liquid ammonia as 1.1, is $1.1 \times 21.2 = 23.3$ B. t. u. per pound.

The available heat, therefore, is $577.8 - 23.3 = 554.5$ B. t. u. per pound.

The weight of ammonia required, then $= \frac{81,900}{554.5} = 148$ pounds per hour or $\frac{148}{250} = 59$ per cent of the plant output.

Therefore, if the plant product is used as the cooling medium in the final condensers, 60 per cent of the output will be in the form of ammonia gas at 25 pounds absolute available for absorption purposes, as for production of aqua or ammoniacal salts and 40 per cent as anhydrous liquid ammonia. Of course, if a separate refrigeration system were installed for the final condensation, 100 per cent of the product would be liquid anhydrous ammonia.

Ammonia Receivers and Weighing Devices

The liquid ammonia receivers are commercial seamless drawn cylinders hung from calibrated springs (Figure 20) by means of which production is weighed. There are two such cylinders per set, arranged in series, although the second one is to take the vapor pressure of the ammonia only. A relief valve set at 500 pounds is in the line with the second cylinder. In operation, the gas at 300 atmospheres containing liquid ammonia enters cylinder 1, deposits the liquid ammonia and passes out. When this cylinder is full, it is dumped into cylinder 2, from which the ammonia can be drawn off to shipping cylinders or the cylinder loading storage
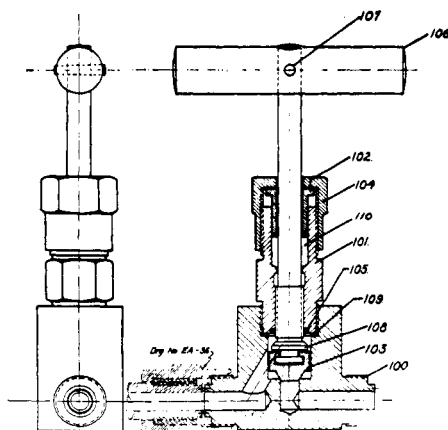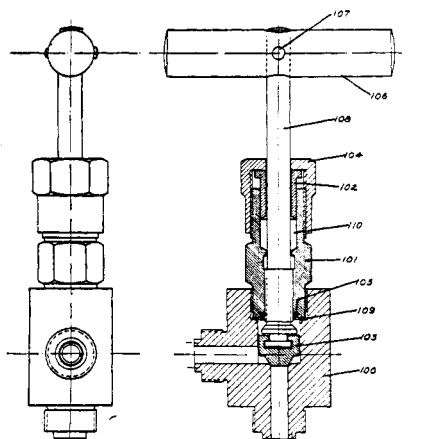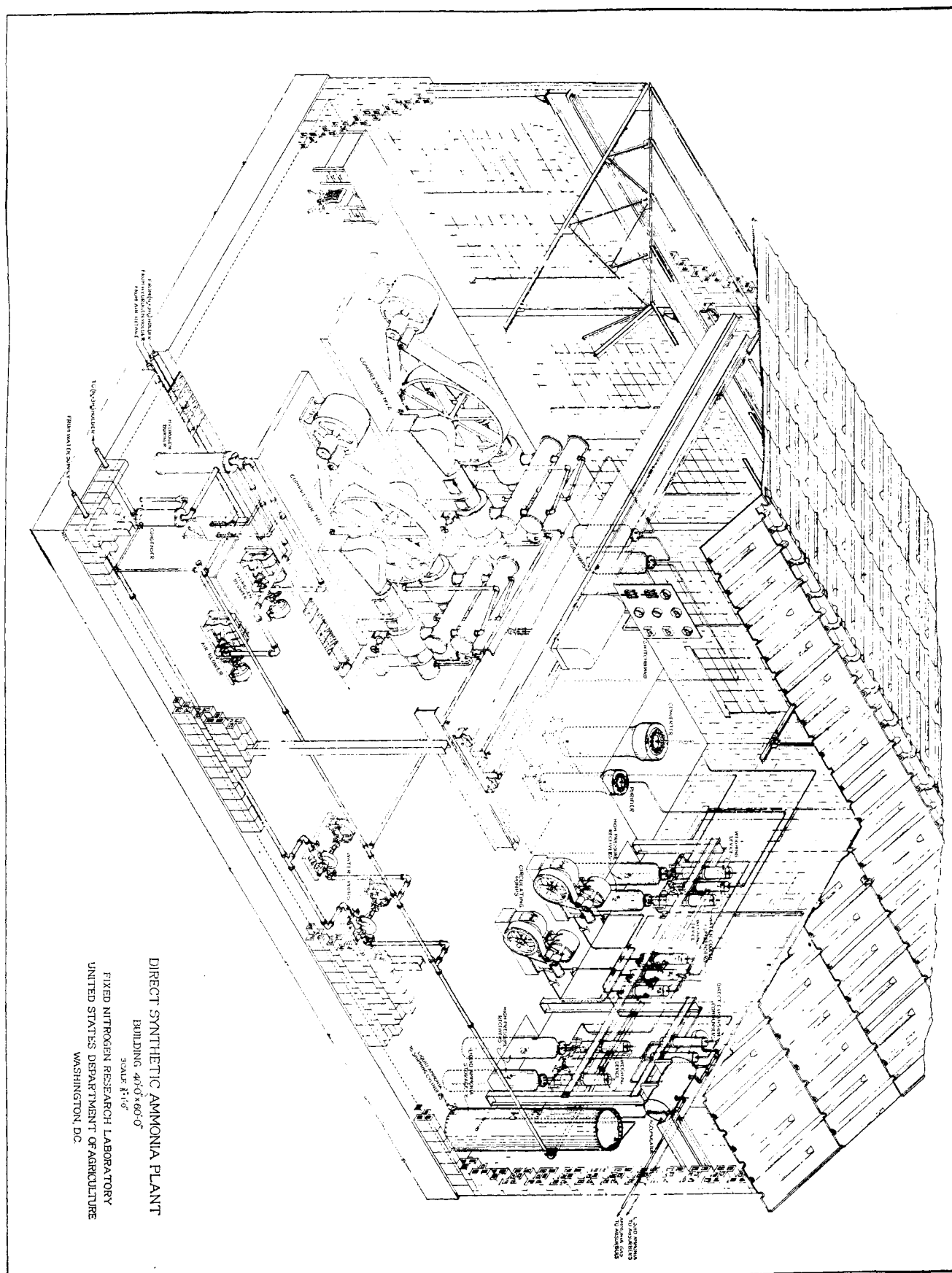

Figure 25—Line Valves

or otherwise as desired. The weight scale for the calibrated spring is so marked off as to show empty when in reality there are 100 pounds of ammonia remaining in the cylinder and registers full when there is still space for 100 pounds additional. An alarm sounds when these empty and full points have been reached.

DIRECT SYNTHETIC AMMONIA PLANT
BUILDING 40'-0" x 60'-0"
SCALE 1/8" = 1'-0"
FIXED NITROGEN RESEARCH LABORATORY
UNITED STATES DEPARTMENT OF AGRICULTURE
WASHINGTON, D.C.

Although the relief valve on cylinder 2 should take care of any building up of pressure in this cylinder, due to a valve leak for instance, and so prevent a serious accident when loading from this to a relatively low-pressure container, further precautions have been taken. Between cylinders 1 and 2 are two line valves and between these valves a line closed by a valve to the atmosphere. Under normal operation with ammonia collecting in cylinder 1 and not being emptied into cylinder 2, the two valves between these cylinders are closed while the third valve is open. A valve leak will now be to the atmosphere, avoiding the possibility of building up the pressure of cylinder 2. When ammonia is to be loaded from cylinder 1 to cylinder 2, the valve to the atmosphere is closed while the other two valves are opened.

Piping, Fittings, and Valves

The high-pressure piping of the system is seamless drawn commercial chrome-vanadium (1.0 per cent Cr, 0.2 per cent V, 0.35 per cent C), 0.5 inch i. d. $\times$ 1 inch o. d. tubing. This tubing, although just twice as expensive per pound as ordinary carbon steel hydraulic tubing, weighs approximately 50 per cent less per foot length than a carbon steel tubing of equal strength, so that per foot length the cost is the same, while owing to the smaller o. d. the fittings for the chrome-vanadium tubing are appreciably less.

Figure 21 shows the union connection. In flanging two lengths of tubing together, part 1 is screwed over the end of one length of pipe, part 3, over the end of the other and with a copper gasket between; the union is made tight by pulling up on part 2. In smaller pieces of equipment, such as the valves and condenser heads, the connection corresponding to part 1 is machined as an integral part of the article, while in the larger pieces, such as the converter head, a connection is tapped to receive a plug, also shown in Figure 21, one end of which is cut to correspond to part 1. Figure 22 shows a special connection for taking off a sample or gage line, while the type of regular run fittings is shown in Figure 23. As a small plant cannot stand the overhead of a maintenance machinist, and as often, even in large plants, it is not convenient completely to dismantle a section of piping to make a change, a hand tool has been worked up to prepare the tubing for the union connection. After the thread has been cut with a hand die, it is necessary to square off the end of the pipe, cut back the thread, and cut the V groove. This is all accomplished by means of the tool shown in Figure 24, where parts 11, 10, and 9, respectively, perform the above-mentioned operations when inserted in the handle, 1, and turned against the work by means of a hand ratchet while being fed to the work by feed screw, 3.

Line shut-off valves, straightway and angle, are as shown in Figure 25. These valves, which cost made up considerably less than any commercial valve found, weigh 30 pounds, while the most satisfactory valve as suggested by the industry would weigh as computed from the drawings, 125 pounds. The valve plug which is hardened makes its seat in the softer body of the valve.

The Plant

A collection of this equipment assembled into plant form might appear as on page 787, where the flow as previously explained for the flow sheet, Figure 1, can be followed through.


A Device for Estimating Corrosion[1]

## By W. R. Fetzer

The Miner Laboratories, Chicago, Ill.

THE chemist is often asked to make comparative corrosion tests of metals and alloys under conditions ranging from the temperature of brine tanks to that of steam sterilization. The results are wanted immediately and at little cost. The method must be such that subsequent tests duplicating the conditions of the first can be made at any time.

The apparatus herein described answers such requirements. It is an enlarged "yeast rocker,"[2] modified to meet present needs. Glass tubes, closed at one end, 30 cm. long and 4 cm. in diameter, are bent at right angles in the middle, slightly flattened on the bottom, and fastened by clips of spring brass to a board 20 cm. (8 inches) wide and somewhat shorter than the water bath. The board is affixed to a length of 6-mm. (0.25-inch) pipe which rests in bearings anchored to each end of the water bath. The frame is rocked from sixteen to twenty times a minute through an angle of 30 to 40 degrees. Small pieces of sheet metal, 24 gage, 4 $\times$ 5 cm., are used for test purposes. They are bent lengthwise at right angles and placed in the tube resting on the edges. The rocker frame is so set that the test pieces remain near the bend of the tube. The lower part of the tube is filled with the corroding liquor. The mouth of the tube is closed loosely with a cork or a plug of cotton. In rocking, the liquid flows into the open part and air bubbles pass into the closed end, and on the return the air is forced out. In this manner the

Test Piece
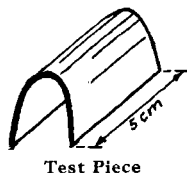
liquid is kept saturated with oxygen, agitation is sufficient to supply the corroding surface with fresh liquid, and erosion is a comparatively small factor.

The tests are generally made at room temperature, although the thermostat may be regulated to meet a specific requirement. The samples are usually weighed every other day and the test is continued until the loss of weight is practically constant. Fresh corroding liquor is added at proper intervals to avoid an accumulation of metal in solution and to approximate more nearly the actual conditions of service.
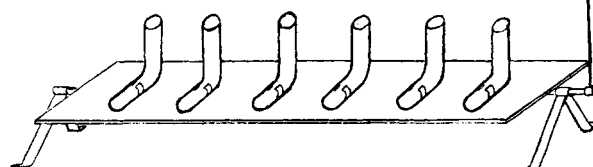
"Rocker" for Determining Corrosion Rate of Metals

The tabulated data, from an experiment conducted at room temperature to determine the corrosive action of 10 per cent vinegar on a number of metal alloys, illustrate the degree of accuracy obtainable by this method.

Corrosive Action of Vinegar on Copper
(Area of test piece 39.1 sq. cm.)

| | \-\-\-\-Loss in Weight\-\-\-\- | | | |
| | Grams | | Mg./sq. dm./day | |
Days	(1)	(2)	(1)	(2)
2	0.0624	0.0618	80	79
4	0.1830	0.1830	117	117
7	0.3936	0.3922	144	143
11	0.5936	0.6086	137	141
15	0.8018	0.8124	137	139
19	1.0222	1.0238	138	140

[1] Received June 10, 1925.
[2] Fraser, J. Phys. Chem., 25, 4 (1921).